

INTERNATIONAL
STANDARD

ISO
5794-1

Second edition
1994-12-15

**Rubber compounding ingredients — Silica,
precipitated, hydrated —**

Part 1:
Non-rubber tests

*Ingrédients de mélange du caoutchouc — Silices hydratées
précipitées*

Partie 1: Essais sur le produit brut



Reference number
ISO 5794-1:1994(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 5794-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 5794-1:1984), which has been technically revised.

ISO 5794 consists of the following parts, under the general title *Rubber compounding ingredients — Silica, precipitated, hydrated*:

- Part 1: *Non-rubber tests*
- Part 2: *Test recipe and determination of physical properties in rubber*

Annexes A, B, C and D form an integral part of this part of ISO 5794. Annex E is for information only.

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Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 1: Non-rubber tests

WARNING — Persons using this part of ISO 5794 should be familiar with normal laboratory practice. This part of ISO 5794 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 5794 specifies methods of test for characterizing precipitated hydrated silica for use as a rubber compounding ingredient. A definition is given.

ISO 5794-2 specifies methods of test for precipitated hydrated silica in compounded rubber.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5794. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5794 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-8:1979, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold extraction method.*

ISO 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 787-10:1993, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method.*

ISO 787-18:1983, *General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure.*

ISO 842:—¹⁾, *Raw materials for paints and varnishes — Sampling.*

ISO 3262:1975, *Extenders for paints.*

ISO 4652-1:1994, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: Single-point procedures.*

1) To be published. (Revision of ISO 842:1984)

3 Definition

For the purposes of ISO 5794, the following definition applies.

3.1 precipitated hydrated silica: Material composed of amorphous particles obtained from soluble silicates by precipitation from aqueous solution.

4 Sampling

Sampling shall be carried out in accordance with ISO 842.

5 Methods of test

The properties of precipitated hydrated silica shall be determined by the methods of test referred to in table 1.

Table 1 — Methods of test

Property	Method of test
Silica content of dried sample, % (<i>m/m</i>)	ISO 3262:1975, clause 17, except that in the expression of results the denominator shall be m_0 , where m_0 is the mass, in grams, of the test portion taken in subclause 11.2 of ISO 3262:1975, not the mass of ignited residue obtained in 11.2
Colour	ISO 3262:1975, clause 7
Residue on sieve (nominal aperture size 45 μm):	
— for silica in powder form	ISO 3262:1975, clause 8
— for silica in other forms	ISO 787-18
Matter volatile at 105 °C (loss on heating)	ISO 787-2 (Use a test portion of 2 g weighed to the nearest 0,1 mg)
Loss on ignition at 1 000 °C of dried sample	ISO 3262:1975, clause 11
pH of slurry	ISO 787-9
Water-soluble matter	ISO 787-8
Total copper content, mg/kg	See annex A
Total manganese content, mg/kg	See annex B
Total iron content, mg/kg	See annex C
Specific surface area, m^2/g	See annex D
Density, Mg/m^3	ISO 787-10

Annex A (normative)

Determination of total copper content

A.1 Principle

A test portion is digested with hydrofluoric acid and sulfuric acid and the silicon is volatilized as silicon tetrafluoride.

Any metals in the digested test portion are dissolved in hydrochloric acid, then the solution is diluted and aspirated into the flame of an atomic absorption spectrometer set at a wavelength of 324,7 nm.

The method is applicable to the determination of copper contents up to 125 mg/kg, and there is provision for extending the range to 1 250 mg/kg.

WARNING — All recognized health and safety precautions shall be taken when performing this method of analysis.

A.2 Reagents and materials

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of equivalent purity.

A.2.1 Acetylene, compressed-gas supply.

A.2.2 Air, compressed-gas supply.

A.2.3 Hydrochloric acid, 10 % (*m/m*) solution.

Dilute 20 cm³ of 35 % (*m/m*) hydrochloric acid solution ($\rho_{20} = 1,18 \text{ Mg/m}^3$) with 50 cm³ of water.

A.2.4 Hydrofluoric acid, 40 % (*m/m*) solution ($\rho_{20} = 1,13 \text{ Mg/m}^3$).

A.2.5 Sulfuric acid, 98 % (*m/m*) solution ($\rho_{20} = 1,84 \text{ Mg/m}^3$).

A.2.6 Nitric acid, 68 % (*m/m*) solution ($\rho_{20} = 1,42 \text{ Mg/m}^3$).

A.2.7 Copper, standard solution corresponding to 1 g of Cu per cubic decimetre.

Dissolve 1,000 g \pm 0,001 g of high-purity copper

turnings in a mixture of 10 cm³ of water and 5 cm³ of nitric acid (A.2.6) in a 100 cm³ beaker. Boil under a fume hood to expel oxides of nitrogen. Cool, transfer to a 1 dm³ volumetric flask, make up to the mark with water and mix.

1 cm³ of this standard solution contains 1 000 μg of copper.

A.2.8 Copper, standard solution corresponding to 50 mg of Cu per cubic decimetre.

Pipette 50,0 cm³ of the 1 g/dm³ standard copper solution (A.2.7) into a 1 dm³ volumetric flask, add 5 cm³ of nitric acid (A.2.6), make up to the mark with water and mix.

1 cm³ of this standard solution contains 50 μg of copper.

A.2.9 Copper, standard solution corresponding to 10 mg of Cu per cubic decimetre.

Pipette 50,0 cm³ of the 50 mg/dm³ standard copper solution (A.2.8) into a 250 cm³ volumetric flask, add 1 cm³ of nitric acid (A.2.6), make up to the mark with water and mix.

1 cm³ of this standard solution contains 10 μg of copper.

NOTE 1 Commercially available standard copper solutions may be used, if preferred, instead of A.2.7, A.2.8 and A.2.9.

A.3 Apparatus

Usual laboratory equipment, plus the following:

A.3.1 Platinum dish, of capacity approximately 35 cm³.

A.3.2 Atomic absorption spectrometer, fitted with an air/acetylene burner.

A.3.3 Analytical balance, capable of weighing to 0,1 mg.

A.4 Procedure

A.4.1 Test portion

Weigh, to the nearest 0,1 mg, approximately 2 g of sample into the platinum dish (A.3.1).

A.4.2 Blank test

Carry out a blank test simultaneously with the determination, using the same reagents and same procedures, but omitting the test portion.

A.4.3 Preparation of the calibration graph

A.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm³ volumetric flasks, transfer the volumes of 10 mg/dm³ standard copper solution (A.2.9) indicated in table A.1, dilute to the mark with water and mix.

Table A.1 — Standard calibration solutions for determination of copper

Volume of standard copper solution (A.2.9) cm ³	Corresponding copper content µg/cm ³
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

A.4.3.2 Spectrometric measurements

Aspirate in turn each of the standard calibration solutions prepared in A.4.3.1 into the flame of the atomic absorption spectrometer (A.3.2) and record their absorbances at a wavelength of 324,7 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

A.4.3.3 Plotting the graph

Plot a graph having, for example, the copper contents, in micrograms per cubic centimetre, as abscissae and the corresponding values of absorbance as ordinates.

A.4.4 Determination

A.4.4.1 Preparation of the test solution

Add 10 cm³ of hydrofluoric acid solution (A.2.4) and 0,5 cm³ of sulfuric acid solution (A.2.5) to the test portion (A.4.1) in the dish (A.3.1).

Place the dish and contents on a heated sand tray and evaporate under a fume hood until the evolution of dense white fumes ceases.

Dissolve any residue in 5 cm³ of hydrochloric acid solution (A.2.3) and transfer to a 10 cm³ volumetric flask, rinsing the dish with two 1 cm³ portions of water and adding the rinsings to the flask. Make up to the mark with water, and transfer the solution to a dry polyethylene bottle.

A.4.4.2 Spectrometric measurements

Aspirate the test solution prepared in A.4.4.1 and the blank test solution (see A.4.2) into the flame of the atomic absorption spectrometer and measure their absorbances at 324,7 nm, following the instructions of the instrument manufacturer. Repeat this procedure and record the mean values of the absorbance of the test solution and the blank test solution.

Aspirate water into the flame after each measurement.

If the absorbance of the test solution is greater than that of the standard calibration solution having the highest copper content, dilute 5 cm³ of the test solution to 50 cm³ with water, repeat the measurements and take the dilution into account in the expression of results.

A.5 Expression of results

By reference to the calibration graph, determine the copper contents corresponding to the absorbances of the test solution and the blank test solution.

Calculate the total copper content of the sample $w(\text{Cu})$, expressed in milligrams per kilogram, from the equation

$$w(\text{Cu}) = \frac{10 (M_1 - M_2)}{m}$$

where

M_1 is the copper content, in micrograms per cubic centimetre, of the test solution;

M_2 is the copper content, in micrograms per cubic centimetre, of the blank test solution;

m is the mass, in grams, of the test portion.

If the test solution was diluted as described in A.4.4.2, multiply the right-hand side of the equation by 10.

Express the result to the nearest 0,1 mg/kg.

A.6 Test report

The test report shall include the following information:

- a) all details required for complete identification of the sample;
- b) a reference to annex A of this part of ISO 5794;
- c) the test conditions;
- d) the result obtained for each sample;
- e) any deviations from the procedure specified which might have affected the results.

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Annex B (normative)

Determination of total manganese content

B.1 Principle

The principle is the same as for the determination of total copper content (see annex A), except that the absorbance of the test solution is measured at 279,5 nm and is compared with the absorbance of standard calibration manganese solutions.

The method is applicable to the determination of manganese contents up to 125 mg/kg, and there is provision for extending the range to 1 250 mg/kg.

WARNING — All recognized health and safety precautions shall be taken when performing this method of analysis.

B.2 Reagents and materials

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of equivalent purity.

B.2.1 Acetylene.

See A.2.1.

B.2.2 Air.

See A.2.2.

B.2.3 Hydrochloric acid solution.

See A.2.3.

B.2.4 Hydrofluoric acid solution.

See A.2.4.

B.2.5 Sulfuric acid solution.

See A.2.5.

B.2.6 Nitric acid solution.

See A.2.6.

B.2.7 Manganese, standard solution corresponding to 1 g of Mn per cubic decimetre.

Dissolve 1,000 g \pm 0,001 g of high-purity, oxide-free manganese in a mixture of 50 cm³ of water and

5 cm³ of nitric acid (B.2.6) in a 400 cm³ beaker. Boil under a fume hood to expel oxides of nitrogen. Cool, transfer to a 1 dm³ volumetric flask, make up to the mark with water and mix.

1 cm³ of this standard solution contains 1 000 μ g of manganese.

B.2.8 Manganese, standard solution corresponding to 50 mg of Mn per cubic decimetre.

Pipette 50,0 cm³ of the 1 g/dm³ standard manganese solution (B.2.7) into a 1 dm³ volumetric flask, add 5 cm³ of nitric acid (B.2.6), dilute to the mark with water and mix.

1 cm³ of this standard solution contains 50 μ g of manganese.

B.2.9 Manganese, standard solution corresponding to 10 mg of Mn per cubic decimetre.

Pipette 50,0 cm³ of the 50 mg/dm³ standard manganese solution (B.2.8) into a 250 cm³ volumetric flask, add 1 cm³ of nitric acid (B.2.6), make up to the mark with water and mix.

1 cm³ of this standard solution contains 10 μ g of manganese.

NOTE 2 Commercially available standard manganese solutions may be used, if preferred, instead of B.2.7, B.2.8 and B.2.9.

B.3 Apparatus

As specified in A.3.

B.4 Procedure

B.4.1 Test portion

See A.4.1.

B.4.2 Blank test

See A.4.2.

B.4.3 Preparation of the calibration graph

B.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm³ volumetric flasks, transfer the volumes of 10 mg/dm³ standard manganese solution (B.2.9) indicated in table B.1, dilute to the mark with water and mix.

Table B.1 — Standard calibration solutions for determination of manganese

Volume of standard manganese solution (B.2.9) cm ³	Corresponding manganese content µg/cm ³
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

B.4.3.2 Spectrometric measurements

Aspirate in turn each of the standard calibration solutions prepared in B.4.3.1 into the flame of the atomic absorption spectrometer and record their absorbances at a wavelength of 279,5 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

B.4.3.3 Plotting the graph

Plot a graph having, for example, the manganese contents, in micrograms per cubic centimetre, as abscissae and the corresponding values of absorbance as ordinates.

B.4.4 Determination

B.4.4.1 Preparation of the test solution

See A.4.4.1.

B.4.4.2 Spectrometric measurements

Aspirate the test solution prepared in B.4.4.1 and the blank test solution (see B.4.2) into the flame of the atomic absorption spectrometer and measure their absorbances at 279,5 nm, following the instructions

of the instrument manufacturer. Repeat this procedure and record the mean values of the absorbance of the test solution and the blank test solution.

Aspirate water into the flame after each measurement.

If the absorbance of the test solution is greater than that of the standard calibration solution having the highest manganese content, dilute 5 cm³ of the test solution to 50 cm³ with water, repeat the measurements and take the dilution into account in the expression of results.

B.5 Expression of results

By reference to the calibration graph, determine the manganese contents corresponding to the absorbances of the test solution and the blank test solution.

Calculate the total manganese content of the sample $w(\text{Mn})$, expressed in milligrams per kilogram, from the equation

$$w(\text{Mn}) = \frac{10 (M_3 - M_4)}{m}$$

where

M_3 is the manganese content, in micrograms per cubic centimetre, of the test solution;

M_4 is the manganese content, in micrograms per cubic centimetre, of the blank test solution;

m is the mass, in grams, of the test portion.

If the test solution was diluted as described in B.4.4.2, multiply the right-hand side of the equation by 10.

Express the result to the nearest 0,1 mg/kg.

B.6 Test report

The test report shall include the following information:

- all details required for complete identification of the sample;
- a reference to annex B of this part of ISO 5794;
- the test conditions;
- the result obtained for each sample;
- any deviations from the procedure specified which might have affected the results.

Annex C (normative)

Determination of total iron content

C.1 Principle

The principle is the same as for the determination of total copper content (see annex A), except that the absorbance of the test solution is measured at a wavelength of 248,3 nm and is compared with the absorbances of standard calibration iron solutions.

The method is applicable to the determination of iron contents up to 125 mg/kg, and there is provision for extending the range to 2 500 mg/kg.

WARNING — All recognized health and safety precautions shall be taken when performing this method of analysis.

C.2 Reagents and materials

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of equivalent purity.

C.2.1 Acetylene.

See A.2.1.

C.2.2 Air.

See A.2.2.

C.2.3 Hydrochloric acid solution.

See A.2.3.

C.2.4 Hydrofluoric acid solution.

See A.2.4.

C.2.5 Sulfuric acid solution.

See A.2.5.

C.2.6 Iron, standard solution corresponding to 1 g of Fe per cubic decimetre.

Dissolve 1,000 g \pm 0,001 g of high-purity iron in a mixture of 10 cm³ of water and 5 cm³ of hydrochloric acid (C.2.3) in a 100 cm³ beaker. Boil under a fume hood until dissolution is complete. Cool, transfer to a 1 dm³ volumetric flask, make up to the mark with water and mix.

1 cm³ of this standard solution contains 1 000 μ g of iron.

C.2.7 Iron, standard solution corresponding to 50 mg of Fe per cubic decimetre.

Pipette 50,0 cm³ of the 1 g/dm³ standard iron solution (C.2.6) into a 1 dm³ volumetric flask, add 5 cm³ of hydrochloric acid (C.2.3), dilute to the mark with water and mix.

1 cm³ of this standard solution contains 50 μ g of iron.

C.2.8 Iron, standard solution corresponding to 10 mg of Fe per cubic decimetre.

Pipette 50,0 cm³ of the 50 mg/dm³ standard iron solution (C.2.7) into a 250 cm³ volumetric flask, add 1 cm³ of hydrochloric acid (C.2.3), make up to the mark with water and mix.

1 cm³ of this standard solution contains 10 μ g of iron.

NOTE 3 Commercially available standard iron solutions may be used, if preferred, instead of C.2.6, C.2.7 and C.2.8

C.3 Apparatus

As specified in A.3.

C.4 Procedure

C.4.1 Test portion

See A.4.1.

C.4.2 Blank test

See A.4.2.

C.4.3 Preparation of the calibration graph

C.4.3.1 Preparation of standard calibration solutions

Into a series of six 50 cm³ volumetric flasks, transfer the volumes of 10 mg/dm³ standard iron solution

(C.2.8) indicated in table C.1, dilute to the mark with water and mix.

Table C.1 — Standard calibration solutions for determination of iron

Volume of standard iron solution (C.2.8) cm ³	Corresponding iron content µg/cm ³
0,5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

C.4.3.2 Spectrometric measurements

Aspirate each of the standard calibration solutions prepared in C.4.3.1 into the flame of the atomic absorption spectrometer and record their absorbances at a wavelength of 248,3 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

C.4.3.3 Plotting the graph

Plot a graph having, for example, the iron contents, in micrograms per cubic centimetre, as abscissae and the corresponding values of absorbance as ordinates.

C.4.4 Determination

C.4.4.1 Preparation of the test solution

See A.4.4.1.

C.4.4.2 Spectrometric measurements

Aspirate the test solution prepared in C.4.4.1 and the blank test solution (see C.4.2) into the flame of the atomic absorption spectrometer and measure their absorbances at 248,3 nm, following the instructions of the instrument manufacturer. Repeat this procedure and record the mean values of the absorbance of the test solution and the blank test solution.

Aspirate water into the flame after each measurement.

If the absorbance of the test solution is greater than that of the standard calibration solution having the highest iron content, dilute 5 cm³ of the test solution to 100 cm³ with water, repeat the measurements and take the dilution into account in the expression of results.

C.5 Expression of results

By reference to the calibration graph, determine the iron contents corresponding to the absorbances of the test solution and the blank test solution.

Calculate the total iron content of the sample $w(\text{Fe})$, expressed in milligrams per kilogram, from the equation

$$w(\text{Fe}) = \frac{10(M_5 - M_6)}{m}$$

where

M_5 is the iron content, in micrograms per cubic centimetre, of the test solution;

M_6 is the iron content, in micrograms per cubic centimetre, of the blank test solution;

m is the mass, in grams, of the test portion.

If the test solution was diluted as described in C.4.4.2, multiply the right-hand side of the equation by 20.

Express the result to the nearest 0,1 mg/kg.

C.6 Test report

The test report shall include the following information:

- all details required for complete identification of the sample;
- a reference to annex C of this part of ISO 5794;
- the test conditions;
- the result obtained for each sample;
- any deviations from the procedure specified which might have affected the results.

Annex D

(normative)

Determination of specific surface area

D.1 Principle

Two flasks of equal volume — one containing the test portion, the other empty — are filled with nitrogen under atmospheric pressure at room temperature. Both flasks are then cooled by immersion in liquid nitrogen.

At this temperature, the test portion adsorbs nitrogen, whereby a pressure difference is created between the flask containing the test portion and the reference flask.

The pressure difference is measured by means of a differential pressure gauge. The specific surface area is calculated from the measured pressure difference, the nitrogen-gas feed pressure and the mass of the test portion.

The method is similar to that described in ISO 4652-1:1994, clause 4 (method B).

WARNING — All recognized health and safety precautions shall be taken when performing this method of analysis.

D.2 Materials

D.2.1 Nitrogen gas in a cylinder, or other source of prepurified nitrogen, of recognized analytical quality.

D.2.2 Liquid nitrogen.

D.3 Apparatus

D.3.1 Adsorption apparatus²⁾ (see figure D.1), comprising a reference flask (G) and a sample flask (H) mounted with gas-tight connections.

These connections are provided with one valve each (A and B) by means of which the flasks can be connected with the atmosphere. The gas to be adsorbed is fed into each flask through the connection pieces.

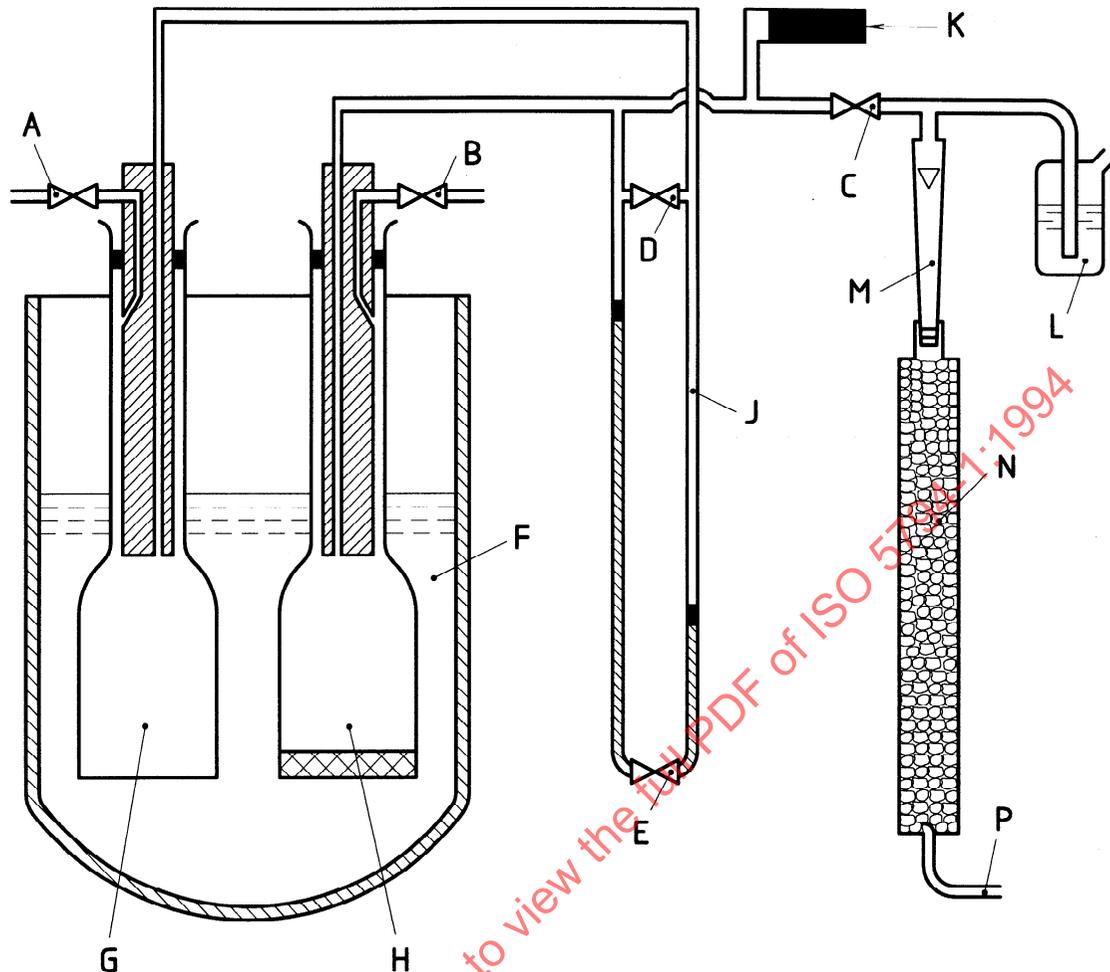
D.3.1.1 The flasks are made of glass which is resistant to sudden changes of temperature and have a volume of approximately 100 cm³. The volume difference between the two flasks shall not exceed 0,1 %.

The flask necks are made of calibrated precision glass tubing with an inside diameter of 5 mm ± 0,02 mm. This ensures that several flasks can be used as sample or reference flasks without having to adjust the compensation volume for each different combination.

D.3.1.2 A U-tube is mounted between the two flasks, and the arms of the pressure gauge are connected by capillary tubes to the two adsorption flasks. By means of valve D, the two adsorption vessels can be either separated from one another or connected to one another via their capillary tubes. Using valve E, the two liquid arms of the differential pressure gauge can be separated or joined together. The measuring fluid is dibutyl phthalate.

The two arms of the differential pressure gauge are made of calibrated precision glass tubing with an inside diameter of 5 mm ± 0,02 mm. Therefore, any change in volume during gas adsorption can be sufficiently accurately calculated. The feed capillary to the sample flask, owing to its short length, is considered as a compensation volume which is adjusted during preparation of the equipment (see D.7).

2) A suitable adsorption apparatus is the Areameter, available from Micromeritics Instrument Corporation, 1 Micromeritics Drive, Norcross, Georgia 30093-1877, USA, and Ströhlein GmbH & Co., Postfach 1460, D-41564 Kaarst, Germany. This information is given for the convenience of users of this part of ISO 5794 and does not constitute an endorsement by ISO of the apparatus named. Other apparatus may be used provided it complies with the specified requirements.



- | | | | |
|--------|-----------------------------|---|---------------------|
| A to E | Valves | K | Compensation volume |
| F | Cold bath | L | Safety valve |
| G | Reference flask | M | Flowmeter |
| H | Sample flask | N | Drying tower |
| J | Differential pressure gauge | P | Nitrogen inlet |

Figure D.1 — Areameter adsorption apparatus

The gas is introduced into the equipment through valve C. If valves A, B and D are open, the gas flows through both flasks. If valves A and D are closed, the reference flask is shut off and only the sample flask is purged with the gas.

When a measurement is made, only part of the volume enclosed by valves A, B and C is cooled to the measurement temperature by the liquid nitrogen. The

remaining volume, which will be at or near room temperature, shall not be more than 10 % of the total volume. The connections to the adsorption flasks are therefore capillaries which almost completely fill the necks of the flasks. In this manner, the volume of gas at room temperature is kept to a minimum.

NOTE 4 Procedures for commissioning new equipment and for carrying out checks are given in D.7.

D.3.2 Control thermostat³⁾, designed to ensure that the adsorption flasks are kept at a constant temperature, and either purged with nitrogen gas or evacuated.

D.3.3 Analytical balance, accurate to 0,1 mg.

D.3.4 Drying oven, capable of being maintained at $105\text{ °C} \pm 2\text{ °C}$.

D.3.5 Cold bath, containing liquid nitrogen (D.2.2).

D.4 Preparation of the test portion

D.4.1 The maximum reading (400 mm) of the differential pressure gauge corresponds to a surface area of approximately 50 m^2 . The mass of the test portion shall therefore be adjusted so that the difference in liquid levels Δh in the differential pressure gauge is as great as possible and at least 50 mm. If the approximate specific surface area is not known, preliminary tests with various test-portion masses shall be performed to establish the most suitable mass for the test portion.

NOTE 5 Table D.1 gives, as a guide, the test-portion masses most suitable for various specific surface areas.

Table D.1 — Test-portion masses suitable for various specific surface areas

Specific surface area m^2/g	Mass of test portion g
20	0,6 to 0,8
30	0,4 to 0,6
40	0,3 to 0,5
80	0,2 to 0,3
120	0,15 to 0,2
140	0,1 to 0,15
> 200	< 0,1

D.4.2 Transfer a suitable amount of silica without predrying to a previously tared sample flask by means of a funnel. Introduce the test portion into the flask in such a manner that no material adheres to the walls of the neck. In order to avoid electrostatic charge

causing the silica to adhere to the neck of the flask, discharge it by putting the flask on a grounded copper plate before and after weighing.

Determine the mass of the test portion by difference, carrying out both weighings to the nearest 0,1 mg.

In order to determine the actual silica content, the mass of the test portion in the flask must be corrected for the moisture content. For this purpose, determine the moisture content immediately after weighing the silica into the Areameter flask, using a separate silica test portion, as follows. Weigh, to the nearest 0,1 mg, 2 g of the silica under test into a weighing bottle with a ground-glass stopper. With the weighing bottle open, dry the test portion for 2 h at $105\text{ °C} \pm 2\text{ °C}$ in the oven (D.3.4). Stopper the hot weighing bottle in the oven and allow it to cool to room temperature in a desiccator. Weigh the stoppered weighing bottle plus contents to the nearest 0,1 mg. Calculate the moisture content of the test portion by difference.

D.4.3 Before determining the surface area, remove as much adsorbed matter as possible from the surface of the test portion by purging in a flow of nitrogen.

Carry out this desorption in a thermostatted enclosure at 150 °C to 160 °C (preferably 155 °C to 160 °C) for a period of $60\text{ min} \pm 5\text{ min}$. Adjust the thermostat thermometer to a height 3 cm above the bottom of the flask. Adjust the nitrogen flow through the flask to $75\text{ cm}^3/\text{min}$.

D.4.4 After desorption, allow the test portion to cool to room temperature under a flow of nitrogen, and stopper and store the flask until required for the determination.

D.5 Procedure

D.5.1 Connect flask H containing the prepared test portion to the nitrogen supply and open valves B and C, allowing nitrogen to flow into the flask with valves D, A and E closed.

D.5.2 Open valves D, A and E and place both reference and sample flask in a water bath maintained at $23\text{ °C} \pm 2\text{ °C}$, maintaining the flow of nitrogen.

3) A suitable thermostat is that for the Areameter, available commercially from Ströhlein GmbH & Co., Postfach 1460, D-41564 Kaarst, Germany. This information is given for the convenience of users of this part of ISO 5794 and does not constitute an endorsement by ISO of the thermostat named. Other thermostats may be used provided they comply with the specified requirements.

D.5.3 After 10 min to 15 min, determine the pressure difference in the flasks by closing valves A, B, C and D. If a pressure difference exists, re-open the valves in the order D, C, B and A and continue purging with nitrogen. Close valves A, B, C and D when the pressures are equal.

D.5.4 When pressure equilibrium has been attained, close valve E and stop the nitrogen flow by closing the supply valves.

D.5.5 Wipe the water drops off the flasks, immerse the flasks in the liquid-nitrogen bath (D.3.5) to the lower mark on the neck and, after 1 min, open valve E very slowly.

D.5.6 When the resulting pressure difference has stabilized, read the difference in heights of the liquid in the U-tube arms to the nearest 0,5 mm.

D.5.7 Close valve E. Replace the liquid-nitrogen bath (D.3.5) with a water bath maintained at approximately 40 °C. After a few minutes, start the nitrogen flow and open valves D, C, B, A and E in that order.

D.5.8 When the connections have reached room temperature, close valves A, D and E and disconnect the sample flask with valves B and C open.

D.6 Expression of results

D.6.1 Method of calculation

Calculate the specific surface area S_m in square metres per gram, using the equation

$$S_m = 1,174 \times 10^{-7} \left[\frac{(1,05 \times 10^5) - p}{m} \right] \times \\ \times \left[(13,71 + 6,656 \times 10^{-5} p) \Delta h + \right. \\ \left. + \frac{m}{\rho} \left(\frac{p}{77,6} - \frac{p_B}{295} \right) \right]$$

where

p is the equilibrium pressure, in pascals, given by

$$p = \frac{109,6 p_B}{393,1 + (9,82 \times 10^{-3} \Delta h)} - 10,29 \Delta h$$

m is the mass, in grams, of the test portion corrected for the moisture content of the sample;

Δh is the difference, in millimetres, in the heights of the liquid in the U-tube arms;

p_B is the atmospheric pressure, in pascals;

ρ is the density, in megagrams per cubic metre, of the test sample, assumed to be equal to 2,0 Mg/m³.

NOTE 6 For specific surface areas greater than 1 m²/g, the term

$$\frac{m}{\rho} \left(\frac{p}{77,6} - \frac{p_B}{295} \right)$$

may be ignored.

Express the result to the nearest 1 m²/g.

D.6.2 Calculation using a nomogram

Calculation may be simplified by the use of a nomogram (see figure D.2).

Place a straightedge over the nomogram to join the measured value of Δh on the Δh scale with the measured value of p_B on the vertical p_B scale. Record the value at the point of intersection of the line and scale A.

Place a straightedge over the nomogram to join the measured value of Δh on the Δh scale with the measured value of p_B on the inclined, reduced p_B scale. Record the value at the point of intersection of the line and scale B.

The specific surface area, in square metres per gram, is given by the equation

$$S_m = \frac{A \Delta h}{m} + \frac{B}{\rho p}$$

where

A and B are the values derived from the nomogram as described above;

Δh , m , ρ and p are as defined in D.6.1.

Express the result to the nearest 1 m²/g.

D.7 Notes on procedure — Preparation of the Areameter

D.7.1 General

When commissioning new equipment or when checking existing equipment, carry out the following tests to check whether the compensation volume (K in figure D.1) is correctly adjusted and that the apparatus is leakproof.

D.7.2 Volume-equalization test

It is essential for this test that valve D is gastight (see D.7.3).

Connect the empty adsorption flasks to the apparatus, aligning the upper marks on the flask necks with the lower gasket. Then open all the valves and purge the apparatus with nitrogen. Use a flow rate of $2,8 \text{ cm}^3/\text{s} \pm 0,15 \text{ cm}^3/\text{s}$.

While purging, keep the flasks immersed in a water bath at $23 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ to the lower mark on the flask necks, so that both flasks attain the same temperature. When this can be assumed to have taken place (allow at least 10 min), seal the apparatus from the atmosphere and separate the flasks from each other by closing valves A, B, C and D in that order. Closing valve D may cause a minor pressure difference. If this pressure difference changes within the next 2 min, complete temperature equilibrium has not been reached between the adsorption flasks. In this case, re-open valves D, C, B and A in that order and again purge the apparatus with nitrogen. Repeat the test after a few minutes. When temperature equilibrium has been reached, close valve E. Remove the water bath, wipe off the adhering water drops and immerse the flasks to the lower mark on the flask necks in a cold bath of boiling nitrogen.

As soon as the flasks have assumed the temperature of the boiling nitrogen (for empty flasks, after approximately 1 min), open valve E very slowly. If the volumes of the sample flask and the reference flask are correctly balanced, there will be no pressure difference between them. In this case, close valve E again and re-open valve D. Then remove the cold bath and replace by a water bath at approximately $40 \text{ }^\circ\text{C}$ to thaw out the flasks. Remove the warm-water bath as

soon as the flasks have reached approximately room temperature. After a few minutes, re-open valves C, B and A in that order, and purge the apparatus with nitrogen.

Cooling and heating the flasks may result in high pressure differences for short periods. This could cause the dibutyl phthalate in the U-tube to enter other parts of the apparatus. It is therefore important that valve E be kept open at the times indicated.

If a pressure difference should arise during the preparation of the apparatus, change the compensation volume (K in figure D.1) at room temperature in such a way that the volumes on both sides of the differential pressure gauge are equal. Check the volume balance by repeating the above test.

D.7.3 Leak test

If balancing the volumes at the temperature of boiling nitrogen results in a continuously increasing, or very considerable, pressure difference (greater than 400 mm) after valve E has slowly been opened, this is evidence of leaks from the apparatus to the atmosphere (i.e. through valves A, B and C).

The gas tightness of valve D cannot be checked by means of the pressure difference, and this valve must therefore be tested separately. To do this, immerse the flasks in the cold bath, close valves B, C and D and open valves A and E. By means of valve A, produce a difference in liquid heights of approximately 300 mm and close valve A.

This difference in height shall not change by more than 1 mm within 10 min.

D.7.4 Operational check

Check the correct operation of the apparatus initially, and periodically during use, by measurement of the surface area of a silica of known specific surface area. The specific surface area of the reference material shall have been measured using the same method.

D.7.5 Maintenance

Replace the dibutyl phthalate in the U-tube if it becomes polluted, or at least once a year. Also replace exhausted silica gel in the drying tower.

D.8 Test report

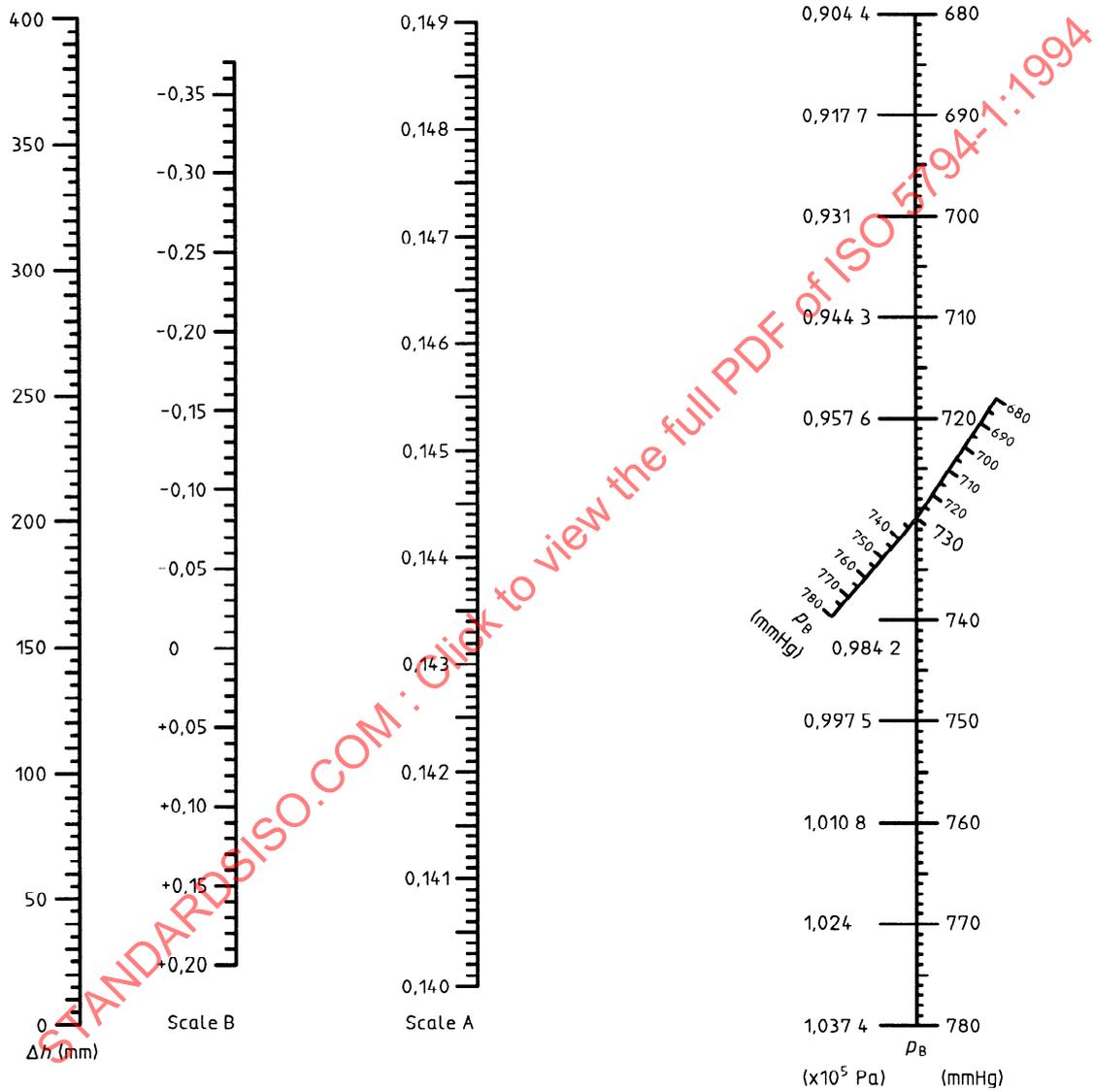
The test report shall include the following information:

- a) all details required for complete identification of the sample;
- b) a reference to annex D of this part of ISO 5794;
- c) the test conditions;
- d) the mass of the test portion used;
- e) the results obtained;
- f) any deviations from the procedure specified which might have affected the results.

Table D.2 — mmHg/kPa conversion table for inclined part of right-hand scale in figure D.2

mmHg	kPa
680	90,6
685	91,3
690	92,0
695	92,6
700	93,3
705	94,0
710	94,6
715	95,3
720	96,0
725	96,6
730	97,3
735	98,0
740	98,6
745	99,3
750	100,0
755	100,6
760	101,3
765	102,0
770	102,6
775	103,3
780	104,0

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NOTE — See table D.2 for conversion of millimetres of mercury into kilopascals.

Figure D.2 — Nomogram

Annex E (informative)

Classification of silicas and typical physical and chemical properties

E.1 General

The purpose of standardizing on a limited number of grades is to avoid a proliferation of grades. Material meeting the requirements given herein is suitable for use in rubber compounds. It does not follow that material not meeting these requirements is unsuitable for such use.

E.2 Classification according to specific surface area

Six grades of silica are defined in table E.1. The method for determining specific surface area is given in annex D.

Table E.1 — Classification of silicas

Grade	Specific surface area m ² /g
A	> 191
B	161 to 190
C	136 to 160
D	106 to 135
E	71 to 105
F	< 70

E.3 Typical values of physical and chemical properties

Typical values of physical and chemical properties, determined by the methods specified in table 1, are given in table E.2.

E.4 Physical form and dispersibility

E.4.1 The physical form of the silica should be such that, when incorporated into the rubber in accordance with the test recipe, and using the equipment and procedure given in ISO 5794-2, the unvulcanized mix so produced has the silica properly dispersed, showing no evidence of agglomerates or uneven dispersion of silica.

E.4.2 To reduce dust levels when the silica is being handled, dust-reduced forms of the powder, such as pellets, granules, beads, etc., are recommended, provided that such forms comply with the dispersibility requirement (see E.4.1) and the other requirements of this part of ISO 5794.

E.5 Rubber tests

Results of tests carried out in accordance with ISO 5794-2 should be as agreed between supplier and consumer.